IS 3522-2 (1989): Textiles - Estimation of common preservatives - Part 2 [TXD 5: Chemical Methods of Test]

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IS 3522 (Part 2) : 1989

Indian Standard

TEXTILES — ESTIMATION OF COMMON PRESERVATIVES — PART 2

(First Revision)

भारतीय मानक

वस्त्रादि—सामान्य परिरक्षियों का आंकलन — भाग 2

UDC 677·047·62 : 677·016·8

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

January 1990

Price Group 3

Chemical Methods of Test Sectional Committee, TDC 5

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards on 31 July 1989, after the draft finalized by the Chemical Methods of Test Sectional Committee had been approved by the Textile Division Council.

This standard was first published in 1970 and has been revised to make it up to date on the basis of experience gained during its use. In this revision the following changes have been carried out.

a) The title and scope have been modified because the methods prescribe the estimation of common preservatives on textiles and not their analysis as such.

b) Method for colorimetric estimation of pentachlorophenyl laurate (PCPL) has been included.

c) Conditioning and testing atmospheres for the test specimens have been included.

d) Method for estimation of 2, 4-ditnitro-1-naphthol and 4, 6-dinitro-ortho-cresol has been modified to include colorimetric estimation.

e) The method for estimation of dieldrin has been deleted because dieldrin is toxic and can cause death within 24 hours, if inhaled. It is also skin irritant. The use of this agent is not recommended for preservation of textiles.

f) The time of extraction of DDT from textiles has been doubled for complete extraction.

g) Method for estimation of mercaptobenzthiazol (MBT) has been deleted as this agent is not used now-a-days for preservation of textiles.

During storage or in use, most of the textile materials are liable to suffer damage as a result of attack by micro-organisms, fungi and bacteria. Numerous treatments have been developed for textile materials with different preservatives (fungicides and insecticides) to protect the material for staining and degradation arising from attack of micro-organisms and insects.

Methods for estimation of the following preservatives have been covered in Part 1 of this standard:

a) Salicylanilide, b) Salicylic acid, c) Pentachlorophenol, d) Sodium silicofluoride, e) zinc chloride, and zinc naphthenate, f) Copper naphthenate, g) Copper-8-hydroxy-quinolin, and h) Copper salcylanilide.

The methods prescribed in this standard are applicable for estimating single known preservative present on yarns and fabrics of different materials, and these are not applicable to mixtures of preservatives. Every precaution should be taken to protect the yarn or fabric being sampled.

In reporting the result of a test or analysis made in accordance with the standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

AMENDMENT NO. 1 JANUARY 1992
TO
IS 3522 (Part 2) : 1989 TEXTILES — ESTIMATION OF COMMON PRESERVATIVES — PART 2

(First Revision)

(This amendment is being issued to incorporate estimation of permethrin, a new mothproofing agent for wool and its blends, developed by Defence Research and Development Establishment, Gwalior, Defence Research and Development Organization, Ministry of Defence, Government of India, after extensive research and field trials.)

(*Page* 1, *clause* **1.1**) — Insert the following at the end of the clause:

f) Permethrin

(*Page* 5, *clause* **10.4.2**) Insert the following new clauses after **10.4.2**:

'**11 ESTIMATION OF PERMETHRIN**

11.1 General

11.1.1 This test method is applicable for estimation of permethrin applied in wool fabrics for its protection from insect damage. Permethrin is extracted from the wool fabric into solution. This extract, at a known dilution is then injected to the Gas Liquid Chromatographic apparatus (GLC) under specified conditions. The result of the analysis is then compared with standard known solution of permethrin, obtained under the same conditions.

11.2 Procedure

11.2.1 Weigh a sample of mothproofed wool fabric of 10 g after conditioning for 12 h at 27°C and 65 percent relative humidity. Transfer it to a Soxhlet extraction apparatus and extract for 6 h at a rate of six solvent exchange per hour with 100 ml of 2-methoxyethanol solvent in round bottom flask. Evaporate the extract to dryness and dissolve the extract in 5 ml of hexane and transfer to 10 ml volumertric flask. Rinse the round bottom flask with 5 ml hexane and transfer it to volumetric flask and make up to the mark.

11.2.2 To prepare standard solutions of known concentrations, dissolve accurately weighed quantity of permethrin (technical) in a suitable volume of isopropanol. From this stock solution prepare standard solutions of desired concentration by dilution with isopropanol in volumetric flask (10 ml).

11.2.3 Inject 2 or 3 μl of standard solutions (obtained in **11.2.2**) and then of extract sample (obtained in **11.2.1**) into the GLC operated under the conditions given in **11.2.4**. Thus obtain the chromatographs for standard solutions (known concentrations) and for the extract sample (unknown).

11.2.4 *Operating Conditions*

11.2.4.1 The operating conditions of the GLC with Flame Ionization Detector shall be as follows:

Column	—	Glass column, 2 percent OV-17 on chromosorb AW DMCS, 1 metre (length) × 3 mm (internal diameter)
Temperatures	—	Oven — 263°C
		Injector — 290°C
		Detector — 290°C
Gas flow rates	—	N_2 — 76 ml/min
		H_2 — 40 ml/min
		Air — 300 ml/min
Attenuation	—	× 8
Chart speed	—	5 mm/min
Retention time	—	250 seconds
Analysis time	—	18 minutes

NOTE — A typical gas chromatographic run showing retention time and shape of the peak is given in Fig. 1.

11.2.5 Measure the peak areas of the standard solutions of permethrin and draw a calibration graph by plotting peak areas against the known concentrations.

11.2.6 Measure the peak areas of the extract sample and convert into a concentration by using calibration graph (obtained in **11.2.5**). Then calculate quantity of permethrin present in the total extract sample (10 ml) (obtained in **11.2.1**).

11.2.7 The percentage of permethrin present on the wool weight is determined by using the following formula;

$$\text{Permethrin, percent (on wool weight)} = \frac{100 \times QPE}{WWF}$$

QPE = calculated quantity of permethrin present in the extract sample, and

WWF = weight of wool fabric taken for extraction.

11.2.8 Repeat the test with the remaining test samples and calculate the percentage of permethrin on the wool sample and then determine the average of all the values.

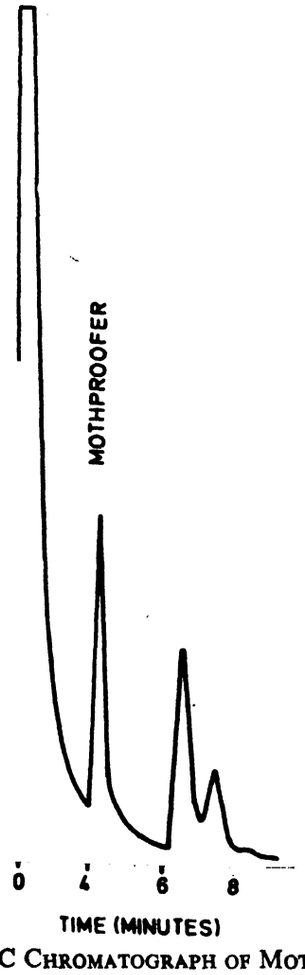

FIG. 1 GC CHROMATOGRAPH OF MOTHPROOFER

(TXD 5)

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Indian Standard

TEXTILES — ESTIMATION OF COMMON PRESERVATIVES — PART 2

(First Revision)

1 SCOPE

1.1 This standard (Part 2) prescribes methods for estimation of the following preservatives on textiles:

a) *p*-nitrophenol

b) 2, 4-dinitro-1-naphthol (DAN)

c) 4, 6-dinitro-ortho-cresol (DNOC)

d) Dichloro-diphenyl-trichloroethane (DDT)

e) Pentachlorophenyl laurate (PCPL)

2 REFERENCES

2.1 The following Indian Standards are necessary adjuncts to this standard:

IS No.	*Title*
IS 1070 : 1977	Specification for water for general laboratory use (*second revision*)
IS 3522 (Part 1) : 1989	Method for estimation of common preservatives used in textile industry — Part 1

3 SAMPLING

3.1 Lot

The quantity of textile material of one definite type and quality delivered to a buyer against one despatch note shall constitute a lot.

3.2 Unless otherwise agreed to between the buyer and the seller, the number of bundles or pieces to be selected at random from a lot shall be in accordance with Table 1 or Table 2, respectively.

Table 1 Sample Size for Yarn
(*Clauses* 3.2 *and* 3.3)

Lot Size (Number of Bundles in the Lot)	Sample Size (Number of Bundles to be Selected)
(1)	(2)
Up to 150	3
151 ,, 300	4
301 ,, 500	5
501 ,, 1 000	7
1 001 ,, 2 000	8
2 001 ,, 10 000	9
10 001 and above	10

Table 2 Sample Size for Fabrics
(*Clauses* 3.2 *and* 3.3)

Lot Size (Number of Pieces in the Lot)	Sample Size (Number of Pieces to be Selected)
(1)	(2)
Up to 100	2
101 ,, 150	3
151 ,, 300	4
301 ,, 500	5
501 ,, 1 000	7

3.3 From each bundle of yarn or piece of fabric selected as in **3.2**, cut out small portions each weighing about 25 g from at least two different parts and mix them. This shall constitute the test sample. While taking the sample, care shall be taken to exclude a sufficient length of yarn or fabrics from both the ends.

4 PREPARATION OF THE TEST SPECIMEN

4.1 Cut the test sample into small pieces. Mix all the pieces thoroughly and draw at least three test specimens from among these pieces each weighing about 1·5 g or as that required in the test.

5 QUALITY OF REAGENTS

5.1 Unless specified otherwise, pure chemicals shall be employed in tests and distilled water (*see* IS 1070 : 1977) shall be used where the use of water as reagent is intended.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the test results.

6 CONDITIONING AND TESTING ATMOSPHERE

6.1 All the test specimens prior to test shall be conditioned to moisture equilibrium from the dry side in the standard atmosphere at 65 ± 2 percent relative humidity and 27 ± 2°C temperature, at least for 24 hours. All the mass determination shall be made in the standard atmosphere after conditioning.

7 ESTIMATION OF PARANITROPHENOL

7.1 Reagents

7.1.1 *Standard p-Nitrophenol Solution*

The solution is prepared by dissolving 0·25 g of p-nitrophenol in 1 ml of 2 N sodium

hydroxide solution and made up to 250 ml with water.

7.1.2 *Acetic Acid*, 2 N.

7.1.3 *Sodium Hydroxide Solution*, 2 N and 0·1 N.

7.1.4 *Ortho-cresol Solution*, 1 percent (*m/m*), freshly prepared in sufficient alkali (0·1 N) (*see* 7.1.3).

7.1.5 *Zinc Dust*

7.2 Procedure

7.2.1 Take a test specimen of about 5 g weighed accurately to the nearest mg.

7.2.2 Extract the test specimen with 100 ml of 0·1 N sodium hydroxide solution for one hour at boil. Cool the extract, filter it and make up the volume to 250 ml.

7.2.3 Take 25 ml of the extract in a flask. Add 10 to 15 ml of 2N acetic acid and 1g of zinc dust. Heat the solution on boiling water-bath for 2 to 3 minutes to initiate the reduction. Allow the solution to remain for one hour at room temperature. Filter the solution into a 100 ml measuring flask. Wash and make up the volume to 100 ml.

7.2.4 Take 10 ml of standard p-nitrophenol solution and treat it as in 7.2.3 and make up the volume to 100 ml.

7.2.5 Take 10 ml of solution obtained as in 7.2.3 and 1 ml of reduced standard p-nitrophenol solution (7.2.4) in two Nessler tubes. Add 5 ml of freshly prepared ortho-cresol solution to each. Make the solution in both the tubes sufficiently alkaline by adding 2 N sodium hydroxide solution. Shake well and allow to stand for one hour till blue colour develops. Add more alkali to ensure complete development of colour. Make up the volume in each Nessler tube to 50 ml and compare the colour visually.

NOTE — It would be necessary to prepare different standard p-nitrophenol solutions in different Nessler tubes for comparison. In case of dispute, optical density method should be prepared. The optical density should be measured at wave-length of 615 nm.

7.3 Calculations

Calculate the percentage of p-nitrophenol by the following formula:

$$P = \frac{A}{M}$$

where

P = percentage, by mass of p-nitrophenol;

A = volume, in ml, of the reduced standard p-nitrophenol solution required to match the colour of the test solution; and

M = mass, in g, of the test specimen.

7.4 Repeat the test with the remaining test specimens and calculate the percentage of *p*-nitrophenol in each test specimen and then determine the average of all the values.

8 ESTIMATION OF 2, 4-DINITRO-1-NAPHTHOL (DAN) AND 4, 6-DINITRO-ORTHO-CRESOL (DNOC)

8.1 General

8.1.1 The method is applicable to the determination of 2, 4-dinitro-1-naphthol and 4, 6-dinitro-ortho-cresol in textile material provided that dyestuffs are absent which are soluble in diethylether. The preservative is extracted from the material by hot extraction with ammonia solution; the solution acidified and the nitrobody extracted with ether. The residue from the other extract is dissolved, reduced and treated with ferric chloride solution. The optical density of the coloured solution is measured on a suitable spectrophotometer at a wavelength of 470 nm.

8.2 Reagents

8.2.1 *Diethylether*

8.2.2 *2, 4-Dinitro-1-naphthol Standard Reference Solutian*

The solution 0.250 g/l is prepared by dissolving 0 250 g 2, 4-dinitro-1-haphthol reagent in 20 ml of sodium hydroxide (0.1 N) solution and diluting with water to 1000 ml. 1 ml ≡ 0.000 25 g.

8.2.3 *4, 6-Dinitro-ortho-cresol Standard Reference Solution*

The solution 0·250 g/l is prepared by dissolving 0·250 g of 4, 6-dinitro-ortho-cresol reagent in 20 ml of sodium hydroxide 0·1 N solution and diluting with water to 1 000 ml. 1 ml ≡ 0·000 25 g.

8.2.4 *Zinc Dust*

8.2.5 *Acetic Acid 5M Reagent Solution*

8.2.6 *Ammonia Solution (0·2 N)*, dilute 5 N reagent solution 25 times.

8.2.7 *Ferric Chloride*, The solution 100 g/l, is prepared by dissolving 10 g of ferric chloride in water and making up to 100 ml.

8.2.8 *Hydrochloric Acid, Concentrated*, 36 percent (*m/m*) (11 N).

8.2.9 *Hydrochloric Acid 5 N Reagent Solution*

8.2.10 *Lead Acetate Solution*

The solution, 100 g/l, is prepared by dissolving 10 g of lead acetate in water and making up to 100 ml.

8.2.11 *Sodium Hydroxide Solution (2·5 N)*, dilute 5 N reagent solution two times.

8.2.12 *Sodium Hydroxide Solution (0·1 N)*, dilute 5 N reagent solution fifty times.

8.3 Procedure

8.3.1 Extract 5·0 g of shreded textile under reflux with 50 ml to 75 ml of water containing 3 ml of 0·2 N ammonia solution.

8.3.2 Decent off the extract and repeat the extraction until the extract is colourless and gives no colour on addition of sodium hydroxide solution. Bulk the separate extract, filter if necessary and add 5 ml of 2·5 N sodium hydroxide reagent solution to extract and evaporate if necessary to about 100 ml. Neutralize the solution to *p*H 6 with acetic acid solution, add 10 ml of lead acetate solution and allow to stand for 1 hour. Filter and wash the precipitate with hot water, keeping the volume of water used for washing as low as possible.

8.3.3 Make the combined filtrate and washings distinctly acidic with hydrochloric acid 5 N reagent solution and extract with 50 ml of diethyl ether until the aqueous layer remains colourless on making alkaline with sodium hydroxide 2·5 N reagent solution. Combine the ether extracts and evaporate the ether to within a few ml of dryness, removing the last traces of ether without application of heat. Dissolve the extracted nitro body in 60 ml of hot water adding 1 ml of 0·1 N sodium hydroxide solution to the solution to prevent loss of nitrobody. Place on a boiling water bath, maintained at the boil, add 1 g of zinc dust and 2 ml concentrated hydrochloric acid in that order and continue heating on the water bath for exactly 15 minutes, stirring occasionally and avoiding loss by spray.

8.3.4 Filter through Whatman No. 42 filter paper into a 250 ml volumetric flask and wash the zinc residue well with water. Add 2·5 ml of ferric chloride solution and make up to 25 ml with water. Mix well. If the solution shows the slightest turbidity it must be refiltered. Measure the optical density of the solution on a suitable spectrophotometer at a wavelength of 470 nm with water as a blank. The calculation of 2, 4-dinitro-1-naphthol (DAN) and 4, 6-dinitro-ortho-cresol (DNOC) content may be made from a previously prepared calibration graph.

8.4 Calibration

8.4.1 To a series of 250 ml beakers, add 20, 40, 60 and 80 ml of the DAN or (DNOC) standard solution. To an empty beaker add 80 ml of water, neutralize each with hydrochloric acid 5 N solution and make all the solutions to approximately 80 ml with water. Heat to the boil and place on a water bath. Maintain at the boil, add to each 1 g of zinc dust and 2 ml hyprochloric acid (concentrated) in that order and continue heating on the water bath for exactly 15 minutes stirring occasionally and avoiding loss by spray.

8.4.2 Filter through Whatman No. 42 filter paper into a 250 ml volumetric flask and wash the zinc residues well with water. Add to each 2·5 ml of ferric chloride solution and make up to 250 ml with water. Mix well.

8.4.3 Measure the optical density of the solution at a wavelength of 470 nm using 5 mm cells with water in the reference cell.

8.4.4 Construct a graph by plotting optical density against concentration of preservative agent. The range covered by the details given is 0·01 to 0·40 percent.

9 ESTIMATION OF DDT

9.1 Reagents

9.1.1 *Ethyl Ether*

9.1.2 *Ethanolic Potassium Hydroxide Solution*, 1 N.

9.1.3 *Acetone*

9.1.4 *Nitric Acid*, 2 N.

9.1.5 *Standard Silver Nitrate Solution*, 0·5 N.

9.1.6 *Ferric Alum*, 10 percent (*m*/*v*).

9.1.7 *Standard Potassium Thiocyanate Solution*, 0·05 N.

9.1.8 *Nitrobenzene*

9.2 Procedure

9.2.1 Draw one test specimen of about 10 g weighed accurately to the nearest mg.

9.2.2 Extract the specimen in the Soxhlet apparatus for four hours with ethyl ether at the rate of 10 to 12 extractions per hour.

9.2.3 Evaporate the ether off the extract and add 50 ml of acetone and 20 ml of 1 N ethanolic potassium hydroxide. Keep at 20 to 25°C for 15 minutes and add 50 ml of distilled water. Add 20 ml of 2 N nitric acid and exactly 25 ml of 0·5 N standard silver nitrate solution. Shake, add 2 ml of nitrobenzene, shake again, add 2 ml of 10 percent ferric alum solution and titrate the standard excess silver nitrate with 0·05 N standard potassium thiocyanate solution.

9.2.4 Carry out a blank titration side by side.

9.3 Calculations

9.3.1 Calculate the percentage of DDT present in the material using the following formula:

$$P = \frac{(V_2 - V_1) \times N \times 100 \times 0.354\,6}{M}$$

where

P = percentage of DDT, by mass in the test specimen;

V_2 = volume, in ml, of potassium thiocyanate solution required for blank titration (*see* **9.2.4**);

V_1 = volume, in ml, of potassium thiocyanate solution required for the titration of excess of silver nitrate solution (*see* **9.2.3**);

N = normality of standard potassium thiocyanate solution, and

M = mass, in g, of the test specimen.

NOTE — Hydrolysable chlorine in DDT is 10 percent by mass.

9.4 Repeat the test with the remaining test specimens and calculate the percentage of DDT in each test specimen and then determine the average of all the values.

10 ESTIMATION OF PENTACHLOROPHENYL LAURATE (PCPL)

10.1 General

The method is applicable to the determination of pentachlorophenyl laurate in the absence of added pentachlorophenol. The proofing is hydrolyzed, acidified and steam distilled and the pentachlorophenol in the distillate extracted with 1, 1, 1-trichloroethane and complexed with copper sulphate-pyridine reagent. The optical density of the complex in 1, 1, 1-trichlorethane is measured on a suitable spectrophotometer at 450 nm. If pentachlorophenol is believed to be present in an amount greater than 10 percent of the amount of pentachlorophenol is believed to be present in an amount greater than 10 percent of the amount of pentachlorophenol laurate then the procedure described should be carried out out in conjuction with that described in **8** of IS 3522 (Part 1) : 1989.

10.2 Reagents

10.2.1 *Ethanediol (ethylene glycol)*

10.2.2 *1, 1, 1-trichloroethane (referred to hereafter as trichloroethane)*

10.2.3 *Pyridine*

10.2.4 *Sodium Hydroxide Pellet*

10.2.5 *Sodium Sulphate, Anhydrous*

10.2.6 *Copper Sulphate Reagent Solution 50 g/l*

10.2.7 *Pentachlorophenol (standard reagent)*, recrystallized melting point 188°C minimum.

10.2.8 *Hydrochloric Acid Concentrated 36 percent (m/m) (11 M)*

10.3 Procedure

10.3.1 Weigh 2·5 ± 0·05 g of the material cut into small pieces of not more than 5 mm square and place in a dry 250 ml round bottom flask (B24/29 socket). Add 30 ml of ethanediol, 4 g of sodium hydroxide (pellet form), 2 ml to 4 ml of water, in that order and a few anti-bumping granules. Connect the flask with a double surface condenser, bring the contents to boiling point on a sand bath and boil them vigorously for 30 minute under reflux.

10.3.2 At the end of this period allow the contents of the flask to cool, remove the reflux condenser and carefully add through a funnel 60 ml of water followed by 20 ml of hydrochloric acid. Steam distill the contents of the flask ensuring that a constant volume is maintained by applying gentle heat as necessary. Collect 300 ml of distillate in a suitable receiver, applying particular care to prevent loss of pentachlorophenol in the distillate by having adequate cooling. Discontinue the external heating of the flask a few minutes before disconnecting the steam supply. Disconnect the condenser and fit it vertically over the distillate receiver. Wash down the condenser with 25 ml to 30 ml of trichloroethane and collect the washings in the distillate. Transfer the distillate and trichloroethane washings to a 500 ml separating funnel and shake thoroughly. Allow the layers of water and trichloroethane to separate completely before running off the trichloroethane layer into a 100 ml separating funnel. Wash the condenser and distillate receiver with a further 25 ml to 30 ml trichloroethane and add this to the aqueous solution in the 500 ml separating funnel. Repeat the extraction as previously described and add the trichloroethane layer to the first trichloroethane extract in the 100 ml separating funnel. Add to the bulked trichloroethane extract 10 ml of copper sulphate-pyridine reagent (prepared by mixing 4 ml of pyridine with 6 ml of copper sulphate solution immediately before use) and shake well. After effecting complete separation of the aqueous and trichloroethane layer run the lower trichloroethane layer into a 100 ml volumetric flask via a small funnel containing anhydrous sodium sulphate supported by means of quartz wool plug. Add a small quantity of trichloroethane to the copper sulphate-pyridine solution remaining in the separating funnel, shake and allow the layers to separate before filtering the trichloroethane layer through the quartz wool, and collect in the volumetric flask. Wash the filter with further small quantities of trichloroethane and finally make up to 100 ml trichloroethane.

10.3.3 Determine the optical density of the solution using a suitable spectrophotometer at a wavelength of 456 nm using trichloroethane as a blank. Estimate the pentachlorophenyl laurate content by reference to a calibration graph prepared from known standard of pentachlorophenol (1·0 percent pentachlorophenol ≡ 1·71 percent pentachlorophenyl laurate.)

NOTE — If the proofing is expected to contain both pentachlorophenol and the ester then the free pentachlorophenol content should be determined as described in IS 3522 (Part 1) : 1989 and the amount found deducted from the apparent pentachlorophenyl laurate content.

10.4 Calibration

10.4.1 *Direct*

Prepare a calibration graph using 5, 10, 15 ml aliquots of a standard solution of pentachlorophenol reagent in trichloroethane (1 g/200 ml) to cover a range of 1, 2 and 3 percent respectively. Dilute each aliquot to 50 ml to 60 ml with trichloroethane, add 10 ml of copper sulphate pyridine reagent, shake well and then follow the described procedure.

Plot optical density against concentration of pentachlorophenyl laurate.

10.4.2 *Indirect*

Prepare a calibration graph using 5, 10 and 15 ml aliquots of a standard solution of pentachlorophenol reagent (1 g/200 ml) in dilute sodium hydroxide (only sufficient hydroxide solution to ensure complete solution of the pentachlorophenol is necessary). Place each aliquot in a round bottomed flask, add 60 ml of water and 20 ml hydrochloric acid. Fit the flask for steam distillation and then follow the described procedure, if the distillation technique is satisfactory then the graph obtained by the procedure described under direct (*see* **10.4.1**) and indirect should be the same.

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